

European Patent Office

Office européen des brevets



EP 0 834 343 A1 (11)

(12)

EUROPEAN PATENT APPLICATION

(43) Date of publication: 08.04.1998 Bulletin 1998/15

(21) Application number: 97117005.5

(22) Date of filing: 30.09.1997

(51) Int. Cl.6: **B01D 39/20**, F01N 3/02, F01N 3/28

(84) Designated Contracting States: AT BE CH DE DK ES FI FR GB GR IE IT LI LU MC NL PT SE Designated Extension States: AL LT LV RO SI

(30) Priority: 03.10.1996 JP 283396/96 12.09.1997 JP 268033/97

(71) Applicant: KABUSHIKI KAISHA TOYOTA CHUO **KENKYUSHO** Aichi-gun Aichi-ken, 480-11 (JP)

(72) Inventors: · Asai, Mitsuru, c/o K.K. Toyota Chuo Kenkyusho Aichi-gun, Aichi-ken (JP)

· Kamiya, Nobuo, c/o K.K. Toyota Chuo Kenkyusho Aichi-gun, Aichi-ken (JP)

· Hohjo, Hiroshi, c/o K.K. Toyota Chuo Kenkyusho Alchi-gun, Alchi-ken (JP)

· Yamamoto, Kolchiro, c/o K.K. Toyota Chuo Kenkyusho Aichi-gun, Aichi-ken (JP)

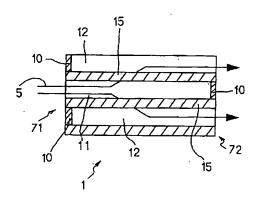
(74) Representative: Blumbach, Kramer & Partner GbR Radeckestrasse 43 81245 München (DE)

Exhaust gas purifying filter (54)

It is an object of the present invention to provide an exhaust gas purifying filter capable of adsorbing soot with a high efficiency and having high burning characteristics.

In the exhaust gas purifying filter, an upstream chamber 11 and a downstream chamber 12 are partitioned from each other by a partitioning wall 15 comprising a porous material having many through-pores. An exhaust gas 5 is introduced into the upstream chamber 11, thus passing through the partitioning wall 15 and flowing into the downstream chamber 12. The total volume of through-pores having diameters in a range of 5 to 20 mm is in a range of 30 to 80% of the total volume of all through-pores of the porous material. The total volume of through-pores having diameters in a range of 20 to 200 um is in a range of 20 to 70% of the total volume of all through-pores of the porous material.

FIG. 1



Description

BACKGROUND OF THE INVENTION

1. Field of the Invention

The present invention relates to an exhaust gas purifying filter and more particularly, to a filter capable of adsorbing soot with a high efficiency and having high burning characteristics.

2. Description of the Related Art

Soot is contained in a gas exhausted from an internal combustion engine such as a diesel engine. Methods of adsorbing the soot by the exhaust gas purifying filter and purifying it have been investigated.

As the exhaust gas purifying filter, a porous material consisting of cordierite, SiC, and the like and having a large number of through-pores formed therein is known. In a monolithic filter of wall flow type which purifies exhaust gas which passes through a partitioning wall, in case of the diameters of through-pores are small when the exhaust gas passes through the filter, a pressure loss of the filter becomes greater, which may deteriorate the performance of the engine. On the other hand, in case of the diameters of the through-pores are large, a part of the soot passes through the partitioning wall of the filter, which deteriorates the soot adsorbing efficiency.

In order to improve the soot adsorbing efficiency and decrease a pressure loss of the filter, an exhaust gas purifying filter is proposed as disclosed in Japanese Laid-Open Patent Publications Nos. 5-23512 and 5-139861. The diameters of pores of the porous material of these exhaust gas purifying filters are adjusted to be $1 - 15\mu m$.

However, the soot adsorbed by the exhaust gas purifying filters accumulates on the surface of the partitioning wall of the porous material of the filter. The accumulated soot is removed by burning it by an electric heater or a burner.

However, the ignition temperature and the burning temperature of the soot adsorbed by this method are high. Thus, the filter consisting of cordierite is partly melted or cracked. Therefore, the method of adsorbing the soot on the surface of the partitioning wall and burning the soot has not yet been adopted in a vehicle widely.

SUMMARY OF THE INVENTION

30

50

The present invention has been devised in view of the above-described problems. Accordingly, it is an object of the present invention to provide an exhaust gas purifying filter capable of adsorbing soot with a high efficiency and having high burning characteristics.

In order to achieve the object, there is provided an exhaust gas purifying filter comprising a porous material having a large number of through-pore formed therein, the large number of through-pores first through-pores of 5 - $20\mu m$ in the total pore volume of the first through-pores occupying 30 - 80% of the total volume of all through-pores of the filter, and second through-pores of 20 - $200\mu m$ (exclusive of 20) in diameter, the total pore volume of the second through-pores occupying 20 - 70% of the total volume of all through-pores of the porous material.

The diameters of the through-pores formed in the porous material are in the above-described range. The diameters of the through-pores in this invention are greater than that of the through-pore formed in the conventional filter on the average. Therefore, when exhaust gas passes through the partitioning wall, soot contained therein is adsorbed not only on the surface of the partitioning wall but also by the through-pores formed in the inside of the partitioning wall. Consequently, the soot contacts the filter in a great area.

Because the soot is adsorbed in a wide area on the surface of the partitioning wall and by the through-pores formed in the inside of the partitioning wall, oxygen contained in the exhaust gas passing through the partitioning wall contacts the soot in a great area. Therefore, the soot adsorbed by the filter can be burnt at a lower temperature, compared with the soot adsorbed by the conventional filter. Further, because the ignition temperature of the soot is low, the soot can be burnt at a low temperature and without generating flame. Accordingly, the filter of the present invention improves the burning characteristic of the soot.

Further, because the partitioning wall of the filter has the above-described pore distribution, it serves as not only the burning place of the soot, but also a reaction place for purifying NOx.

If the diameters of through-pores are not distributed in the above-described range, i.e., if a large number of throughpores having small diameters is formed in the porous material, the soot accumulates on the surface of the partitioning wall, which prevents the soot from being adsorbed by the pores inside the wall. Consequently, the pressure loss of the filter becomes greater and the performance of an engine deteriorates.

On the other hand, if a large number of through-pores having large diameters is formed in the porous material, much soot is exhausted together with exhaust gas. As a result, the soot may be not adsorbed efficiently.

This and other objects, features and advantage of the present invention will become apparent upon reading of the



following detailed description and drawings.

BRIEF DESCRIPTION OF THE DRAWINGS

- 5 Fig. 1 is an explanatory view explaining an exhaust gas purifying filter according to embodiment 1 of the present invention:
 - Fig. 2 is a view showing a method of measuring a pressure loss of the exhaust gas purifying filter of embodiment 1; Fig. 3 is a view showing the burning characteristic of the soot adsorbed by the exhaust gas purifying filter of embodiment 1 and a comparative example 1;
- Fig. 4 is a view showing a adsorbed state of the soot adsorbed by the exhaust gas purifying filter of embodiment 1; Fig. 5 is a view showing an adsorbed state of the soot adsorbed by the exhaust gas purifying filter of comparative example 1;
 - Fig. 6 is an explanatory view showing an exhaust gas purifying filter according to embodiment 2 of the present invention:
- Fig. 7 is a view showing a method of measuring a pressure loss of an exhaust gas purifying filter, according to embodiment 4, installed on a vehicle;
 - Fig. 8 is an explanatory view showing the shape of a through-pore formed straight in a porous material of the exhaust gas purifying filter of the present invention;
 - Fig. 9 is an explanatory view showing the shape of a through-pore zigzag in the porous material of the exhaust gas purifying filter of the present invention;
 - Fig. 10 is an explanatory view showing the shape of a through-pore irregular in its diameter in the porous material of the exhaust gas purifying filter of the present invention;
 - Fig. 11 is an explanatory view showing the distribution of the pores in the porous materials of embodiments 1, 5, and 6 and comparative examples 2 and 3; and
- 25 Fig. 12 is a diagram showing the distribution of the pores in the porous materials of embodiment 1 and comparative example 1.

DETAILED DESCRIPTION OF THE INVENTION

20

35

The through-pores formed in an exhaust gas purifying filter 1 comprising a porous material may have various shapes shown in Figs. 8, 9, and 10. For example, the through-pore 19 shown in Fig. 8 is uniform almost in the diameter thereof and extends almost straight. The through-pore 19 shown in Fig. 9 is uniform almost in the diameter thereof and zigzag. The through-pore 19 shown in Fig. 10 is irregular in its diameter.

The diameter of the through-pore is measured by mercury porosimetory.

It is preferable that the porous material is honeycomb-shaped.

In the exhaust gas purifying filter of the present invention, a porous material is used as a partitioning wall for allowing exhaust gas to pass therethrough.

The diameters of the through-pores of the porous material are not limited to the range of 5 - 20µm and 20 - 200µm. Through-pores having diameters other than the above-described ranges may also be formed in the porous material.

It is preferable that the total volume of through-pores having diameters smaller than 5μm or greater than 200μm is 20% or less of the total volume of all through-pores. The porous material having through-pores whose diameters are in this range adsorbs soot at a high efficiency and is allowed to have an improved soot-burning characteristic.

If the total volume of through-pores having diameters smaller than $5\mu m$ or greater than $200\mu m$ is more than 20% of the total volume of all through-pores, the porous material adsorbs the soot at a low efficiency and is not allowed to have an improved soot-burning characteristic.

The partitioning wall is divided into two layers, namely, a porcus material of upstream side (first layer) and a porcus material of downstream side (second layer). It is preferable that the total volume of through-pores, of the first layer, having diameters in the range of 20 - 200 μ m is set to 60% or more of the total volume of all through-pores of the first layer. It is preferable that the total volume of through-pores, of the second layer, having diameters in the range of 5 - 20 μ m is set to 70% or more of the total volume of all through-pores of the second layer.

In the distribution of the through-pores of the entire partitioning wall, the total volume of through-pores having diameters in the range of 5 - 20µm is in a range of 30 - 80% of the total volume of all through-pores of the porous material; and the total volume of through-pores having diameters in the range of 20 - 200µm is in a range of 20 - 70% of the total volume of all the through-pores of the porous material.

According to the above-described way of distribution of the through-pores, the diameters of the through-pores of the porous material at the upstream side are great, whereas the diameters of the through-pores of the porous material at the downstream side are small. This construction allows the soot to be adsorbed by the partitioning wall at the upstream side in which large-diameter through-pores are formed, thus improving the soot adsorbing efficiency and the

burning characteristic of the adsorbed soot.

It is preferable that the thickness of the first layer is set to 20 - 98% of the entire thickness of the partitioning wall and that the thickness of the second layer is set to 2 - 80% of the entire thickness thereof. If the thickness of the first layer is less than 20% of the entire thickness of the partitioning wall or if the thickness of the second layer is more than 80% of the entire thickness thereof, the soot accumulates on the surface of the partitioning wall, thus clogging the pores. Consequently, the burning efficiency of the soot may deteriorate. If the thickness of the first layer is more than 98% of the entire thickness of the partitioning wall or if the thickness of the second layer is less than 2% of the entire thickness thereof, the soot passes through the partitioning wall at a high percentage. Consequently, the soot adsorbing efficiency may deteriorate.

If the total volume of through-pores having diameters in the range of 20 - $200\mu m$ is less than 60% of the total volume of all through-pores of the first layer, a large number of through-pores have diameters in the range of less than $20\mu m$ or more than $200\mu m$. If a large number of through-pores having diameters less than $20\mu m$ are formed in the first layer, the soot may accumulate on the surface of the partitioning wall. Consequently, the degree of the pressure loss of the filter increases and the burning characteristic of the soot may deteriorate. If a large number of through-pores having diameters more than $200\mu m$ is formed in the first layer, the burning characteristic of the soot may deteriorate.

If in the second layer, the total volume of through-pores having diameters in the range of 5 - $20\mu m$ is less than 70% of the total volume of all through-pores of the second layer, more through-pores have diameters in the range of less than $5\mu m$ or more than $20\mu m$. If a large number of through-pores having diameters less than $5\mu m$ is formed in the second layer, the degree of the pressure loss of the filter may increase. If a large number of through-pores having diameters more than $20\mu m$ is formed in the second layer, the soot adsorbing efficiency may deteriorate.

It is preferable to provide a heat conduction control substance with the surface of the porous material and the surface of the through-pores to improve the heat conductivity thereof. Consequently, the burning efficiency of the soot can be improved. It is preferable that the heat conduction control substance has a higher heat conductivity than the substance of the porous material to improve the burning efficiency of the soot. As the heat conduction control substance, SiC, AIN, Al₂O₃, BeO and the like can be used.

It is preferable that the porous material and the through-pores carry catalyst on the surface thereof to improve the burning characteristic of the soot. As the catalyst, a combination of a noble metal and an oxide can be used. As the noble metal, palladium (Pd), platinum (Pt), rhodium (Rh) and the like can be used. As the oxide, cerium oxide, praseodymium oxide, samarium oxide and the like can be used. It is possible to use a non-stoichiometric oxide consisting of cerium oxide, praseodymium oxide, samarium oxide from which oxygen has been partially removed or alternatively a solid solution of other elements.

The above-described catalyst may be directly carried on the surface of the porous material made of cordierite or SiC or directly on the wall surfaces of the through-pores. It is also possible to coat the surface of the porous material and wall surfaces of the through-pores with powder of an oxide such as Al₂O₃, SiO₂, TiO₂ or ZrO₂ and then apply the catalyst to the powder of the oxide. It is also possible to apply a mixture of the powder of the catalyst and the powder of Al₂O₃, SiO₂, TiO₂ or ZnO₂ to the surface of the porous material.

The heat conduction control substance may be applied to the surface of the partitioning wall (porous material) of the filter and the wall surfaces of the through-pores, and then the catalyst may be carried on the heat conduction control substance. It is also possible to apply a mixture of the heat conduction control substance and/or the catalyst and oxide powder to the surface of the porous material.

EMBODIMENT

45

10

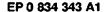
EMBODIMENT 1 and COMPARATIVE EXAMPLE 1

An exhaust gas purifying filter according to an embodiment of the present invention is described below with reference to Figs. 1 through 5.

As shown in Fig. 1, in an exhaust gas purifying filter 1 (hereinafter referred to as filter 1) of embodiment 1, an upstream chamber 11 and a downstream chamber 12 are partitioned from each other by a partitioning wall 15 made of a porous material with many pores. In order to purify an exhaust gas 5, the exhaust gas 5 introduced into the upstream chamber 11 passes through the partitioning wall 15, thus being flowing into the downstream chamber 12.

The cylindrical filter 1 has a diameter of 30mm and a length of 50mm and comprises a plurality of honeycomb-shaped cells in the lengthwise direction of the filter 1. Each cell is open at its one end. One of the cells composes the upstream chamber 11 open at an inlet 71 of the filter 1, whereas the outlet 72 of the upstream chamber 11 is sealed with a sealer 10. The other cells compose the downstream chambers 12 open at the outlet 72 thereof, and the inlet 71 of each downstream chamber 12 is sealed with the sealer 10.

The upstream chamber 11 and the downstream chamber 12 are adjacent to each other through the partitioning wall 15. The exhaust gas 5 is introduced into the upstream chamber 11, thus passing through the partitioning wall 15.



At this time, soot contained in the exhaust gas 5 is adsorbed by the partitioning wall 15.

As a result, the soot contained in the exhaust gas 5 is purified. Then, the purified exhaust gas 5 flows into the downstream chamber 12 and exhausted from the outlet 72.

The method of manufacturing the filter 1 will be described below.

20

40

First, in order to allow a molded product to have the composition of cordierite, kaolin, talc, and alumina were mixed with each other in a ball-mill at a predetermined mixing ratio for 12 hours to obtain 49wt% of powdery mixture. The particle diameters of these substances were 1μm or less. Then, 13wt% of binder (trade name: Celander), 20wt% of graphite powder (diameter: 50 - 300μm) serving as a pore-forming material, and 18wt% of water were added to the mixture. The resulting mixture was kneaded.

Then, a honeycomb-shaped product having a diameter of 35mm and a length of 55mm was obtained by extrusion molding.

After binder-removal, the product was sintered atmosphere at 1440°C for two hours, and the end of each cell was sealed with a sealer. Then the product is subjected to heat treatment. As a result, a filter comprising a cordierite porous material having a large number of pores was obtained.

A filter of comparative example 1 comprising a porous material having pores whose diameters were all less than $30\mu m$ was manufactured in the same manner as embodiment 1 except that 20wt% of graphite powder having diameters of 10 - $30\mu m$ was additionally used as a pore-forming material.

Thereafter, the diameters of the pores of the porous material of embodiment 1 and comparative example 1 were measured by the mercury porosimetory. The result is shown in Fig. 12.

As indicated in Fig. 12, in the distribution of the through-pores of the filter of embodiment 1, the total volume of through-pores having diameters in the range of 5 - 20 μ m was 40% of the total volume of all through-pores of the porous material. The total volume of through-pores having diameters in the range of 20 - 200 μ m was 50% of the total volume of all the through-pores of the porous material. The total volume of through-pores having diameters less than 5 μ m or more than 200 μ m was 10% of the total volume of all through-pores of the porous material.

In the distribution of the through-pores of the filter of comparative example 1, through-pores having diameters greater than 30µm were not present in the porous material, and the average of all the through-pores of the filter of comparative example 1 was 15µm.

The burning characteristics of soot adsorbed by the filter 1 of embodiment 1 and the filter of comparative example 1 were evaluated.

As shown in Fig. 2, the burning characteristics of the soot were measured by a soot burning testing device 3. After the filter 1 adsorbed soot generated by the burning of light oil, air 51 was introduced into the filter 1 from the inlet 71 while the temperature of the air 51 was being raised and exhausted from the outlet 72. The temperature of the air 51 was measured by a thermometer 30 installed at the inlet 71. A detector 31 for detecting the presence of CO and CO₂ was installed at the outlet 72. The burning characteristics of the soot were evaluated according to the amount of the CO and CO₂ generated by the burning of the soot. Supposing that the total amount of the generated CO and CO₂ was 5ppm, the ignition temperature of the soot was 380°C.

The result of the measurement of the burning characteristics of the soot is shown in Fig. 3.

As indicated in Fig. 3, the soot adsorbed by the filter 1 of embodiment 1 started to burn at 380°C.

The soot adsorbed by the filter of comparative example 1 started to burn at 480°C.

The result indicates that the burning temperature of the soot adsorbed by the filter of embodiment 1 is lower by about 100°C than that of the soot adsorbed by the filter of comparative example 1.

Then, the burning speeds of the soot adsorbed by the filters of embodiment 1 and comparative example 1 were measured. In measuring the burning speed of the soot, after the same amount of soot was adsorbed by the filters of embodiment 1 and comparative example 1, the air 51 was introduced into each filter from the inlet 71 while the temperature of the air 51 was being raised. In order to examine the burning speed of the soot, the time when the total amount of the generated CO and CO₂ became 400ppm in relation to the time when the soot started to burn thereof was measured.

The result was that the soot adsorbed by the filter of embodiment 1 was burnt about twice as fast as that adsorbed by the filter of comparative example 1.

The state of the soot adsorbed by both filters were examined by observing the sections thereof.

The result was that as shown in Fig. 4, in the case of the filter 1 of embodiment 1, the soot 6 was adsorbed not only on the surface of the partitioning wall 15 of the filter 1 but also in the interior of through-pores 19 formed through the partitioning wall 15. In particular, a large amount of the soot 6 was adsorbed in the through-pores 19 positioned proximately to the upstream chamber 11, as shown by oblique cross lines of Fig. 4.

In the case of the filter 1 of comparative example 1, as shown by the right oblique lines in Fig. 5, soot 96 was adsorbed by the partitioning wall 95 only on its surface positioned alongside of the upstream chamber 11 and no soot 96 was adsorbed in the interior of through-pores 99.

The soot adsorbing efficiencies of the filters of embodiment 1 and comparative example 1 were measured.

The soot adsorbing efficiency was measured by a smoke meter installed at the downstream side of the filter.

The result was that the soot adsorbing efficiency of the filter of embodiment 1 was 95%, whereas that of the filter of comparative example 1 was 98%. The difference of 3% in the adsorbing efficiency between embodiment 1 and comparative example 1 was so slight that any problems do not occur in the use of the filter 1.

The pressure loss of each filter of embodiment 1 and comparative example 1 was measured.

In measuring the pressure loss of each filter, a pressure gauge was installed at the inlet and the outlet of each filter. Air containing soot was introduced into the filter to measure the air pressure by both pressure gauges. In order to measure the pressure loss, the relative pressure difference, namely, the air pressure at the outlet relative to the air pressure at the inlet was calculated.

The result was that the pressure loss of the filter of embodiment 1 was lower than that of comparative example 1 by about 20%. Thus, the filter of embodiment 1 is useful in use.

EMBODIMENT 2

15

As shown in Fig. 6, the filter 2 of embodiment 2 has a two-layer structure. That is, a partitioning wall 16 of the filter is composed of an upstream part 161 confronting the upstream chamber 11 and a downstream part 162 confronting the downstream chamber 12.

In the distribution of the through-pores in the upstream part 161, the total volume of through-pores having diameters in the range of $20 - 200\mu m$ was 90% of the total volume of all through-pores of the porous material (first layer) of the filter. In the distribution of the through-pores in the downstream part 162, the total volume of through-pores having diameters in the range of $5 - 20\mu m$ was 90% of the total volume of all through-pores of the porous material (second layer) of the filter.

The method of manufacturing the filter will be described below.

Initially, a honeycomb-shaped cordierite porous material was obtained in the same manner as embodiment 1 except that the diameters of particles of graphite as a pore-forming material were 5 - 30 µm.

Then, the porous material was dipped in slurry, and the slurry was attached to the downstream part 162 of the porous material. The slurry was prepared as follows: 0.85wt% of polycarboxyl ammonium serving as dispersant, 20wt% of graphite powder (particle diameter: 20 - 300 µm) serving as a pore-forming material, and 25wt% of water was added to 50.65wt% of cordierite whose particle diameter was 0.5µm. The contents were kneaded for dispersing the contents for 12 hours by a ball mill. Thereafter, 3.5wt% of polyvinyl alcohol (PVA) serving as hardening resin was added to the solution. Then, the contents were kneaded for dispersing the contents therein to prepare the slurry.

Then, the slurry attached to the downstream part 162 of the porous material was dried and binder-removed.

Then, the porous material was sintered at 1400° for two hours in the atmosphere. As a result, as shown in Fig. 6, the filter 2 having a two-layer structure was manufactured. That is, the filter 2 had the upstream part 161 having throughpores whose diameters were in the range of 20 - $200\mu m$ and the downstream part 162 having through-pores whose diameters were in the range of 5 - $20\mu m$.

The burning characteristic of soot was evaluated by the same method as that described in embodiment 1. Most of soot was adsorbed by the upstream part 161 having the larger pores (diameters: $20 - 200 \mu m$). The ignition temperature of the soot adsorbed by the filter 2 was as low as 380°C. The burning speed of the soot adsorbed by the filter 2 was about twice as fast as that of the soot adsorbed by the filter of comparative example 1.

The pressure loss of the filter 2 of embodiment 2 was almost the same as that of the filter 1 of embodiment 1. The soot adsorbing efficiency of the filter 2 of embodiment 2 was also almost the same as that of the filter of comparative example 1.

5 EMBODIMENT 3

The filter of embodiment 3 is the same as that of embodiment 1 except that catalyst was carried on the surface of the partitioning wall and through-pores.

As the catalyst, a cerium oxide was used. In manufacturing the filter of embodiment 3, initially, honeycomb-shaped cordierite porous material was obtained by a method similar to that of embodiment 1. Then, the porous material was dipped in a solution of a cerium oxide nitrate. Thereafter, the porous material was dried at 500°C for two hours in the atmosphere to precipitate solid a cerium oxide on the surface of the partitioning wall partitioning the filter into the upstream chamber and the downstream chamber and the interiors of the through-pores. The amount of the a cerium oxide carried on the surface of the partitioning wall and the pores was 10% of the weight of the filter.

The burning characteristics of the soot adsorbed by the filter carrying the a cerium oxide was evaluated by a method similar to that of embodiment 1. As a result, the ignition temperature of the soot adsorbed by the filter carrying the a cerium oxide thereon was 350°C, whereas the ignition temperature of the soot adsorbed by the filter 1 of embodiment 1 not carrying the a cerium oxide thereon was 380°C.

That is, the soot adsorbed by the filter carrying the a cerium oxide thereon is ignited at a temperature lower by 30°C than the soot adsorbed by the filter not carrying the a cerium oxide thereon.

This effect can be also obtained when powder prepared from a cerium oxide is applied to the porous material of the filter.

The burning characteristic of the soot adsorbed by the filter carrying palladium or platinum thereon in addition to the a cerium oxide was evaluated. The result was that the ignition temperature of the soot adsorbed by such a filter was as low as 320 - 330°C.

This indicates that the soot adsorbed by the filter carrying the a cerium oxide and palladium, or platinum thereon is allowed to have an improved burning characteristic.

EMBODIMENT 4

10

20

35

45

In embodiment 4, the filter was installed on a vehicle to measure a soot-adsorbed state and the change in the load to be applied to the engine thereof under the condition of practical use.

The filter of embodiment 4 was manufactured by a method similar to that of embodiment 1. The outer diameter of the filter was 140mm and its length was 150mm. As shown in Fig. 7, the filter 1 was installed in the rear of a diesel engine 81. A pressure gauge 82 was installed at the inlet 71 of the filter and the outlet 72 thereof. A thermometer 86 was mounted on the inlet 71. The burning characteristic of the soot adsorbed by the filter was measured by changing the load to be applied to the diesel engine 81.

The result was as follows. When a light loading was applied to the diesel engine 81 and the temperature of exhaust gas was low, soot was adsorbed by the filter, and the pressure loss of the filter was great. When a heavy loading was applied thereto and the temperature of the exhaust gas become high, the soot adsorbed by the filter started to burn and the pressure loss thereof became constant. This is because the amount of the soot exhausted from the diesel engine 81 became equal to the burning amount of the soot adsorbed by the filter. The temperature at the inlet 71 was 430°C when the pressure loss became constant.

The burning characteristic of the soot adsorbed by the filter of comparative example 1 having through-pores whose diameters were 1 - $30\mu m$ was measured by a method similar to that of embodiment 4. The result was that when the pressure loss of the filter became constant, the temperature of the inlet of the filter was 520° C.

That is, the soot adsorbed by the filter of embodiment 4 started to burn at the temperature lower by about 90°C than that adsorbed by the filter of comparative example 1. Therefore, the filter of embodiment 4 has an improved burning characteristic.

EMBODIMENT 5

In manufacturing the filter of embodiment 5, in order to allow a molded product to have the composition of the cordierite, kaolin, talc, and alumina were mixed with each other at a predetermined mixing ratio to obtain 49wt% of powdery mixture. Then, 13wt% of binder (trade name: Celander), 15wt% of graphite powder (diameter: 50 - 200µm), 5wt% of graphite powder (diameter: 200 - 300µm), and 18wt% of water were added to the mixture and kneaded. Thereafter, a porous material was obtained by a method similar to that of embodiment 1.

In the distribution of the through-pores of the filter of embodiment 5, the total volume of through-pores having diameters in the range of 5 - $20\mu m$ was 70% of the total volume of all through-pores of the porous material. The total volume of through-pores having diameters in the range of 20 - $200\mu m$ was 25% of the total volume of all through-pores of the porous material. The total volume of through-pores having diameters less than $5\mu m$ or more than $200\mu m$ was 5% of the total volume of all through-pores of the porous material.

The burning characteristic of the filter of embodiment 5 was measured by a method similar to that of embodiment 1. The ignition temperature of the filter of embodiment 5 was 400°C, and the soot adsorbing efficiency was 95%.

EMBODIMENT 6

In manufacturing the filter of embodiment 6, a porous material was obtained in the same manner as embodiment 1 except that 5wt% of graphite powder (diameter: $50 - 200 \mu m$) and 15wt% of graphite powder (diameter: $200 - 300 \mu m$) were used as the pore-forming material.

In the distribution of the through-pores of the filter of embodiment 6, the total volume of through-pores having diameters in the range of 5 - $20\mu m$ was 35% of the total volume of all through-pores of the porous material. The total volume of through-pores having diameters in the range of 20 - $200\mu m$ was 60% of the total volume of all through-pores of the porous material. The total volume of through-pores having diameters less than $5\mu m$ or more than $200\mu m$ was 5% of the total volume of all through-pores of the porous material.

The burning characteristic of the soot adsorbed by filter of embodiment 6 was measured by a method similar to that



of embodiment 1.

5

20

35

40

45

50

55

The soot ignition temperature of the filter of embodiment 6 was 380°C, and the soot adsorbing efficiency was 93%.

COMPARATIVE EXAMPLE 2

In manufacturing the filter of comparative example 2, a porous material was obtained in the same manner as embodiment 1 except that 2wt% of graphite powder (diameter: $50 - 200 \mu m$) and 18wt% of graphite powder (diameter: $200 - 300 \mu m$) were used.

In the distribution of the through-pores of the filter of comparative example 2, the total volume of through-pores having diameters in the range of 5 - $20\mu m$ was 20% of the total volume of all through-pores of the porous material. The total volume of through-pores having diameters in the range of 20 - $200\mu m$ was 70% of the total volume of all through-pores of the porous material. The total volume of through-pores having diameters less than $5\mu m$ or more than $200\mu m$ was 10% of the total volume of all through-pores of the porous material.

The burning characteristic of the filter of comparative example 2 was measured by a method similar to that of embodiment 1.

The soot ignition temperature of the filter of comparative example 2 was 440°C, and the soot adsorbing efficiency was 80%.

COMPARATIVE EXAMPLE 3

In manufacturing the filter of comparative example 3, a porous material was obtained in the same manner as embodiment 1 except that 18wt% of graphite powder (diameter: $50 - 200\mu m$) and 2wt% of graphite powder (diameter: $200 - 300\mu m$) were used.

In the distribution of the through-pores of the filter of comparative example 3, the total volume of through-pores having diameters in the range of $5 - 20 \,\mu m$ was 85% of the total volume of all through-pores of the porous material. The total volume of through-pores having diameters in the range of $20 - 200 \,\mu m$ was 10% of the total volume of all through-pores of the porous material. The total volume of through-pores having diameters less than $5 \,\mu m$ or more than $200 \,\mu m$ was 5% of the total volume of all through-pores of the porous material.

The burning characteristic of the filter of comparative example 3 was measured by a method similar to that of embodiment 1.

The soot ignition temperature of the filter of comparative example 3 was 470°C, and the soot adsorbing efficiency was 95%.

Table 1 shows the soot adsorbing and the soot burning characteristics of the filters (embodiments 1, 5, and 6 and comparative examples 1 through 3).

TABLE 1

5-20 μ m;40%		
	380°C	95%
20-200 μ m;50%		
less than 5 μ m or more than 200 μ m;10%		
E 1 30 μ m or less; 100%	480°C	98%
E 2 5-20 μ m;20%	440°C	80%
20-200 μ m;70%		
less than 5 μ m or more than 200 μ m;10%		
E 3 5-20 μ m;85%	470°C	95%
20-200 μm;10%		
less than 5 μ m or more than 200 μ m;5%		
5-20 μ m;70%	400°C	95%
20-200 μ m;25%		
less than 5 μ m or more than 200 μ m;5%		
5-20 μ m;35%	380°C	93%
20-200 μ m;60%		
less than 5 μ m or more than 200 μ m;5%		
	200 μ m;10% E 1 30 μ m or less; 100% E 2 5-20 μ m;20% 20-200 μ m;70% less than 5 μ m or more than 200 μ m;10% E 3 5-20 μ m;85% 20-200 μ m;10% less than 5 μ m or more than 200 μ m;5% 5-20 μ m;70% 20-200 μ m;25% less than 5 μ m or more than 200 μ m;5% 5-20 μ m;35% 20-200 μ m;60% less than 5 μ m or more than 200 μ m;5%	200 μ m;10% E 1 30 μ m or less; 100% 480°C E 2 5-20 μ m;20% 440°C 20-200 μ m;70% less than 5 μ m or more than 200 μ m;10% E 3 5-20 μ m;85% 470°C 20-200 μ m;10% less than 5 μ m or more than 200 μ m;5% 5-20 μ m;70% 400°C 20-200 μ m;25% less than 5 μ m or more than 200 μ m;5% 5-20 μ m;5% 380°C 20-200 μ m;60% less than 5 μ m or more than

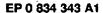
Fig. 11 shows the distribution of through-pores of the respective filters.

The table 1 and Fig. 11 indicate that the ignition temperature of soot is low and the soot is adsorbed at a high percentage when the total volume of through-pores having diameters in the range of $5 - 20 \, \mu m$ is in the range of 30 - 80% of the total volume of all through-pores of the porous material and when the total volume of through-pores having diameters in the range of $20 - 200 \, \mu m$ is in the range of 20 - 70% of the total volume of all the through-pores of the porous material.

While the invention has been described with reference to embodiments, it is to be understood that modification or variations may be easily made by a person of ordinary skill in the art without departing from the scope of the invention which is defined by the appended claims.

Claims

- 1. An exhaust gas purifying filter comprising a porous material having a large number of through-pores formed therein, the large number of through-pores comprising first through-pores of 5 to 20μm in diameter, the total pore volume of the first through-pores occupying 30 to 80% of the total volume of all through-pores of the filter, and second through-pores of 20 to 200μm(exclusive of 20) in diameter, the total pore volume of the second through-pores occupying 20 to 70% of the total volume of all through-pores of the porous material.
- 2. The exhaust gas purifying filter according to claim 1, wherein the large number of though-pores further comprises third through-pore of smaller than 5μm or greater than 200μm of the total volume occupies less than 20% of the total volume of all through-pores.



- 3. The exhaust gas purifying filter according to claim 1, wherein the porous material is disposed as a partitioning wall for allowing an exhaust gas to pass therethrough.
- 4. The exhaust gas purifying filter according to claim 3, wherein the partitioning wall comprises a first layer positioned at an upstream chamber side and a second layer positioned at a downstream chamber side;

5

15

30

35

40

45

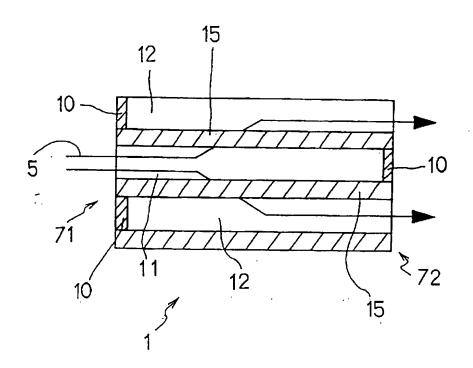
50

55

the total volume of second through-pores in the first layer occupying 60% or more of the total volume of all through-pores of the first layer; and the total volume of the first through-pores in the second layer occupying 70% or more of the total volume of all through-pores of the second layer.

- 5. The exhaust gas purifying filter according to claim 4, wherein the thickness of the first layer occupies 20 to 98% of the entire thickness of the partitioning wall, and the thickness of the second layer occupies 2 to 80%.
 - The exhaust gas purifying filter according to claim 1, wherein a surface of the porous material and wall surfaces of the through-pores are provided with a heat conduction control substance for improving the heat conductivity of the porous material.
 - The exhaust gas purifying filter according to claim 6, wherein the heat conduction control substance is at least one selected from the group consisting of SiC, AIN, Al₂O₃, and BeO.
- 20 8. The exhaust gas purifying filter according to claim 1, wherein a surface of the porous material and wall surfaces of the through-pores are provided with catalyst.
- 9. The exhaust gas purifying filter according to claim 8, wherein the catalyst is a combination of at least one noble metal selected from the group consisting of palladium, platinum, and rhodium and at least one oxide catalyst selected from the group consisting of a cerium oxide, a samarium oxide and a praseodymium oxide.

FIG. 1



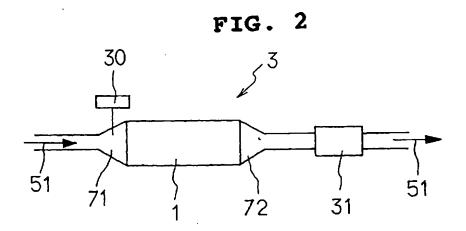


FIG. 3

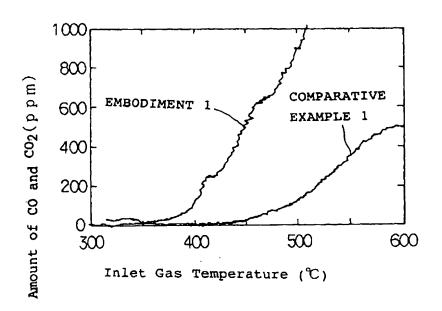


FIG. 4

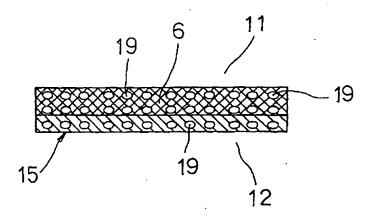


FIG. 5

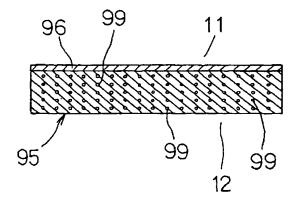
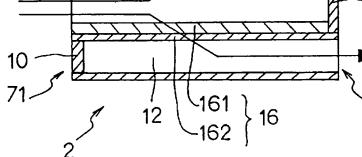


FIG. 6

161 \ 162 \ 16 \ 12

-10



10

FIG. 7

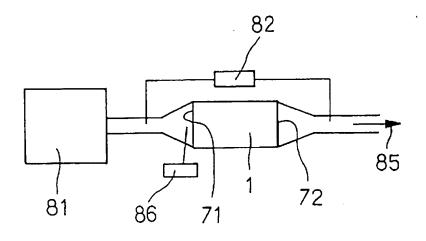


FIG. 8

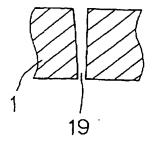


FIG. 9

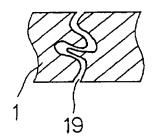


FIG. 10

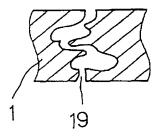


FIG. 11

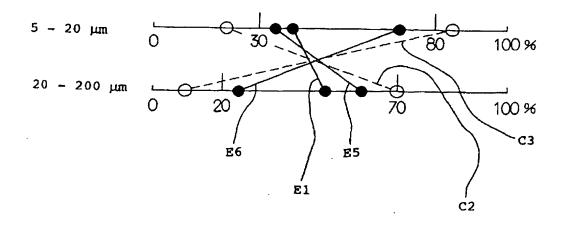
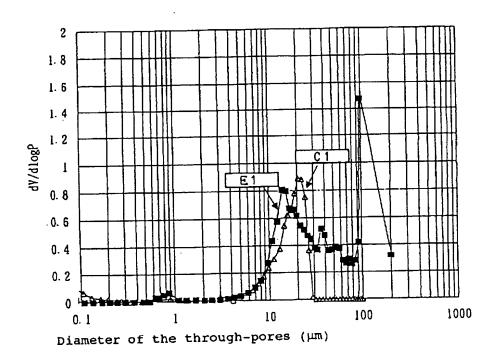


FIG. 12



Distribution of Through-pores

E1 : Embodiment 1

C1: Comparative Example 1



EUROPEAN SEARCH REPORT

Application Number EP 97 11 7005

1	DOCUMENTS CONSIDE				
Category	Citation of document with indi of relevant passag	cation, where appropriate,	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)	
X.	US 4 464 185 A (TOMI * column 2, line 42 figures 1-4 *	TA MASAHIRO ET AL) - column 5, line 37;	1-3,7	B01D39/20 F01N3/02 F01N3/28	
x	DE 35 41 372 A (NIPP * page 9, line 15 - * page 16, line 31 - figure 4 *	page 12, line 24 *	1-3,6,7		
P,X Y	EP 0 736 503 A (NIPP	ON DENSO CO)	1-3,6-8 4,5		
	* the whole document	*			
Y	EP 0 532 986 A (KLOE	CKNER HUMBOLDT DEUTZ	4,5		
	* claims 1-5; figure	: *			
A .	EP 0 630 677 A (ASAM * claims 1-12; figur	HI GLASS CO LTD) re 1; examples 1-4 *	1-4,6,7	,	
A	EP 0 336 883 A (STOR * claims 1-14; examp	BBE PER) Dies 1-3 *	1-3,6-9	SEARCHED (INCC.D)	
A	1	IC ENERGY AUTHORITY	9	B01D F01N B01J	
ı	* claims 1,13,15,16	* 			
	The present search report has	ceen drawn up for all claims			
	Place of search	Date of completion of the search		Examiner	
	BERLIN	15 January 199		Cubas Alcaraz, J	
Y:p	CATEGORY OF CITED DOCUMENTS articularly relevant if taken alone articularly relevant if combined with anot becoment of the same category	E ; earler paten after the filing her D ; dooument cit L : dooument cit	ed in the applicated for other reason	dien ons	
0:r	schnological background ion-written disclosure stermediate dooument	& : member of the document	i. member of the same patent family, corresponding document		

18

This Page is Inserted by IFW Indexing and Scanning Operations and is not part of the Official Record

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

BLACK BORDERS

IMAGE CUT OFF AT TOP, BOTTOM OR SIDES

FADED TEXT OR DRAWING

BLURRED OR ILLEGIBLE TEXT OR DRAWING

SKEWED/SLANTED IMAGES

COLOR OR BLACK AND WHITE PHOTOGRAPHS

GRAY SCALE DOCUMENTS

LINES OR MARKS ON ORIGINAL DOCUMENT

REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY

IMAGES ARE BEST AVAILABLE COPY.

OTHER:

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.